



## **Quality Assurance Project Plan Addendum**

### **Discoverer PM<sub>2.5</sub> Speciation Monitoring Program**

**Shell Offshore, Inc.  
Anchorage, Alaska**

**April 2012**

**Version 1.1**



# **Quality Assurance Project Plan Addendum**

*for the*

## **Discoverer PM<sub>2.5</sub> Speciation Monitoring Program**

**Anchorage, Alaska**

*Prepared for*

**Shell Offshore, Inc.  
Anchorage, Alaska**

By

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April 2012

**A. PROJECT MANAGEMENT ELEMENTS**

**A1 APPROVALS**



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April 20, 2012

Date



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April 20, 2012

Date

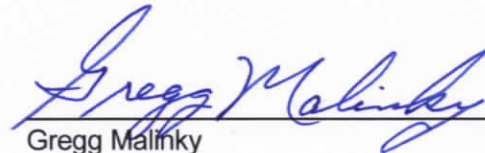


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Gregg Malinky

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Appendix A	Terms, Definitions, Abbreviations
Appendix B	Project References
Appendix C	Project Forms
Appendix D	Standard Operating Procedures

### A3 DISTRIBUTION LIST

Table A-1 provides the names, professional titles, organizations, and contact information of the individuals involved in the development of this Quality Assurance Project Plan (QAPP). This list will be used to distribute this QAPP and any significant revisions.

**Table A-1: QAPP Distribution List**

Name of Individual/Title	Organization	Contact Information
Susan Childs Alaska Venture Support Integrator	Shell Offshore, Inc.	3601 C Street, Suite 1000 Anchorage, AK 99503 (907) 646-7112 Susan.Childs@shell.com
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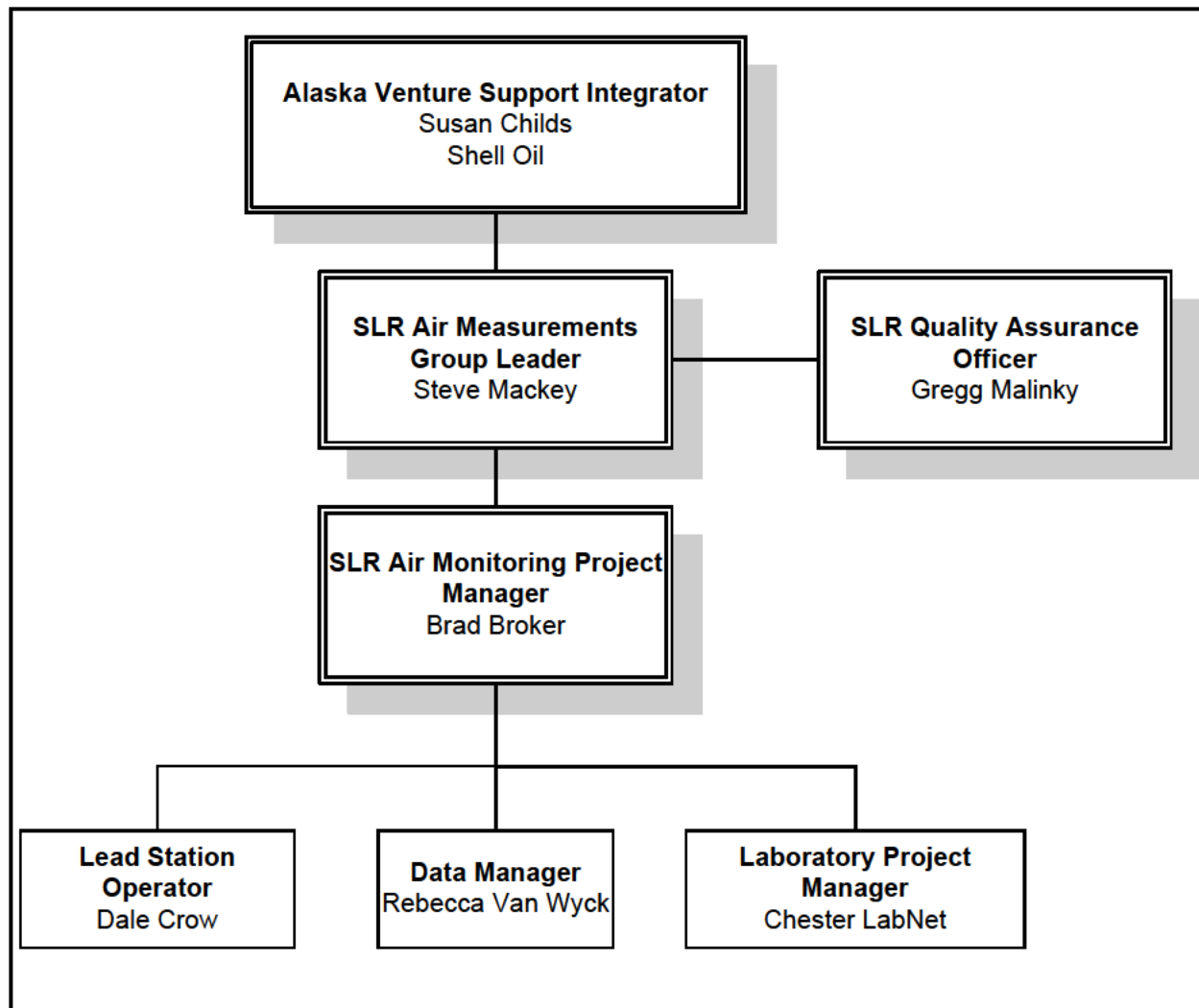
## **A4 PROJECT/TASK ORGANIZATION**

In 2011, Shell Offshore, Inc. (SOI) contracted the services of SLR International Corporation (SLR) to provide PM<sub>2.5</sub> speciation monitoring required to support offshore exploration drilling in the Beaufort and Chukchi Seas. Figure A-1 provides the organization plan for this project. The key personnel involved with this project and their respective responsibilities are summarized in Table A-2. SLR will be responsible for all aspects of air monitoring, database management, and reporting.

**Table A-2: Key Individuals and Responsibilities**

<b>Key individuals</b>	<b>Responsibilities</b>
Alaska Venture Support Integrator	Susan Childs has the overall responsibility for coordination and implementation of the PM <sub>2.5</sub> Speciation Monitoring Project.
SLR Alaska Air Quality Group Manager	Al Trbovich will advise project manager and auditor on data quality assurance and quality control requirements.
SLR Air Measurements Group Leader	Steve Mackey is responsible for the SLR Air Measurements Group organization and leadership. Mr. Mackey will ensure that SLR assets and resources are available to the Project Manager to ensure successful completion of the project objectives.
SLR Project Manager	Brad Broker is responsible for management of the technical staff conducting the PM <sub>2.5</sub> Speciation Monitoring Program and ensuring that the project is completed within established schedules and budgets.
SLR Quality Assurance Officer	Gregg Malinky is responsible for monitoring the implementation of QA/QC procedures and programs to maintain high quality data. Gregg remains organizationally independent of the analysis and distribution processes.
Station Lead Operator	Dale Crow is responsible for overseeing the installation and operation of the PM <sub>2.5</sub> Speciation samplers.
Data Manager	Rebecca Van Wyck is responsible for maintaining the project databases for all field data and for preparing data reports.





**Figure A-1: Monitoring Program Organizational Chart**

## A5 PROBLEM DEFINITION/BACKGROUND AND PROJECT OBJECTIVE

SOI applied for an outer continental shelf (OCS) / prevention of significant deterioration (PSD) air quality permit from the United States Environmental Protection Agency (EPA) in January 2010. The air quality permit is required for the operation of an oil exploratory vessel in the seas north of the Alaska coastline. On January 27, 2012 EPA issued air quality permit R10OCS/PSD-AK-2010-01 to operate the Noble Discoverer (Discoverer) drillship in the Beaufort Sea and air quality permit R10OCS/PSD-AK-09-01 to operate in the Chukchi Sea. The Chukchi Sea permit was issued to Shell Gulf of Mexico Inc. a subsidiary of Shell Oil.

Conditions specified within each of these air quality permits require SOI to conduct post-construction ambient air quality and meteorological monitoring. The air quality permit conditions specify that  $PM_{2.5}$  mass,  $PM_{2.5}$  chemical speciation, horizontal wind direction and speed, temperature, solar radiation, and temperature difference measurements are to be collected.

SLR International Corp (SLR) operates ambient air and meteorological monitoring stations on behalf of SOI at Kaktovik, Alaska and Point Lay, Alaska. Additionally, SLR operates an ambient air and meteorological monitoring station at Wainwright and Deadhorse, Alaska as part of a Shell / ConocoPhillips joint monitoring venture. EPA approved the Quality Assurance Project Plans (QAPP) for Point Lay, Wainwright, and Deadhorse ambient air quality and meteorological monitoring programs on October 14, 2011. The QAPP for the Kaktovik ambient air quality and meteorological monitoring program was approved by EPA on November 16, 2011.

Point Lay and Wainwright ambient air quality and meteorological monitoring stations are located along the Chukchi Sea while the Deadhorse and Kaktovik monitoring stations are located along the Beaufort Sea. Accordingly, data collected at these sites provides the required ambient air quality and meteorological monitoring specified in the SOI permits with the exception of the  $PM_{2.5}$  chemical speciation monitoring. Refer to the approved project QAPPs for specifics on the ambient air quality and meteorological monitoring programs operated at each station.

Permit condition R1.2 (Chukchi permit R10OCS/PSD-AK-09-01) and S1.2 (Beaufort permit R10OCS/PSD-AK-2010-01) each specify: *"The Permittee shall use a continuous sampler and a manual sampler to measure  $PM_{2.5}$ . In addition, filters from the manual sampler shall be analyzed as provided for in the EPA-approved ambient air quality and meteorological plan required pursuant to Condition R.3 to allow for the chemical speciation of  $PM_{2.5}$  constituents, including but not limited to sulfates, nitrates, organics, metals, sea salt, and crustal matter."*

To satisfy these permit conditions, samples collected from the manual samplers operated at Deadhorse were considered. However, these samplers collect samples on a single, Teflon® sample media as part of the  $PM_{2.5}$  mass monitoring program. This Teflon® sample media is only appropriate for a portion of the analyses typically performed to collect  $PM_{2.5}$  chemical speciation data.

Supplemental filter-based samplers capable of collecting samples on multiple filter media are required in order to successfully analyze for all the desired PM<sub>2.5</sub> constituents. Supplemental samplers will be installed at Deadhorse and Wainwright monitoring stations to collect PM<sub>2.5</sub> speciation samples. Deadhorse sampling will satisfy the PM<sub>2.5</sub> speciation monitoring requirements in the Beaufort Sea and Wainwright sampling will satisfy the PM<sub>2.5</sub> speciation monitoring requirements in the Chukchi Sea.

This QAPP describes the PM<sub>2.5</sub> chemical speciation monitoring plan and is considered a supplement to the ambient air quality and meteorological monitoring QAPPs already approved for Point Lay, Wainwright, Deadhorse and Kaktovik air monitoring programs. When used in combination, these QAPPs represent the monitoring plan required in permit condition R.3. (Chukchi permit R10OCS/PSD-AK-09-01) and S.3 (Beaufort permit R10OCS/PSD-AK-2010-01).



Figure A-2: Project Location Map.

## **A6 PROJECT/TASK DESCRIPTION**

Quality Assurance requirements for PM<sub>2.5</sub> Chemical Speciation monitoring are not addressed in any of the published EPA guidance that typically directs PSD ambient air and meteorological monitoring. Absent specific requirements, the SOI PM<sub>2.5</sub> speciation monitoring program is based in part on the EPA Speciation Trends Network (STN) PM<sub>2.5</sub> speciation monitoring program and the Interagency Monitoring of Protected Visual Environments (IMPROVE) program.

PM<sub>2.5</sub> chemical speciation monitoring is required by permit condition R1.2 (Permit R10OCS/PSD-AK-09-01) and S1.2 (R10OCS/PSD-AK-2010-01). Accordingly, PM<sub>2.5</sub> chemical speciation monitoring will continue in both the Chukchi and Beaufort Seas for a minimum of 1 year after commencement of initial operation of the Discoverer as an OCS source.

In 2012, PM<sub>2.5</sub> speciation sampling will occur at Wainwright to satisfy Chukchi Sea monitoring requirements and at Deadhorse to satisfy Beaufort Sea monitoring requirements. Chukchi Sea monitoring may be performed at Point Lay and Beaufort Sea monitoring may be performed at Kaktovik in future years.

### **A6.1 FIELD MEASUREMENTS**

#### **A6.1.1 PM<sub>2.5</sub> Chemical Speciation**

PM<sub>2.5</sub> Chemical Speciation monitoring equipment will be operated using Prevention of Significant Deterioration (PSD) quality assurance practices as a guide. Samplers will be installed, calibrated, and operated according to manufacturer recommended procedures. Calibration and periodic flow-rate verifications will be documented throughout the monitoring program. Field blanks and laboratory quality assurance procedures are described in section B5. Table A-3 lists the parameters that will be measured.

Table A-3: Chemical Speciation Parameters

Parameter / Species	Symbol	Analysis Method	AQS Parameter
Mass	PM <sub>2.5</sub>	Gravimetry	88502
Sulfate	SO <sub>4</sub> <sup>=</sup>	Ion Chromatography <sup>1</sup>	88403
Nitrate	NO <sub>3</sub> <sup>-</sup>	Ion Chromatography <sup>1</sup>	88306/88310
Chloride	Cl <sup>-</sup>	Ion Chromatography <sup>1</sup>	88203
Ammonium	NH <sub>4</sub> <sup>+</sup>	Ion Chromatography <sup>1</sup>	88301
Potassium	K <sup>+</sup>	Ion Chromatography <sup>1</sup>	88303
Sodium	Na <sup>+</sup>	Ion Chromatography <sup>1</sup>	88302
Organic Carbon	OCR	Thermal/Optical Reflectance <sup>2</sup>	88370
Organic Carbon (TOR)	OCT	Thermal/Optical Transmission <sup>2</sup>	88355
OC-Peak 1	OC1	Thermal/Optical Reflectance <sup>2</sup>	88374
OC-Peak 2	OC2	Thermal/Optical Reflectance <sup>2</sup>	88375
OC-Peak 3	OC3	Thermal/Optical Reflectance <sup>2</sup>	88376
OC-Peak 4	OC4	Thermal/Optical Reflectance <sup>2</sup>	88377
Pyrolized Carbon (TOR)	OPR	Thermal/Optical Reflectance <sup>2</sup>	88378
Pyrolized Carbon (TOT)	OPT	Thermal/Optical Transmission <sup>2</sup>	88388
Elemental Carbon (TOR)	ECR	Thermal/Optical Reflectance <sup>2</sup>	88380
Elemental Carbon (TOT)	ECT	Thermal/Optical Transmission <sup>2</sup>	88357
EC-Peak 1	EC1	Thermal/Optical Reflectance <sup>2</sup>	88383
EC-Peak 2	EC2	Thermal/Optical Reflectance <sup>2</sup>	88384
EC-Peak 3	EC3	Thermal/Optical Reflectance <sup>2</sup>	88385
Total Carbon	TC	Thermal/Optical Reflectance <sup>2</sup>	

<sup>1</sup> Analytical measurement determined by NIOSH 7903 / EPA 300.0 Ion chromatography method

<sup>2</sup> Analytical measurement determined by IMPROVE-A carbon analysis method.

**Table A-4: Chemical Speciation Elemental Parameters**

<b>Element <sup>1</sup></b>	<b>Symbol</b>	<b>Element <sup>1</sup></b>	<b>Symbol</b>
Sodium	Na	Germanium	Ge
Magnesium	Mg	Arsenic	As
Aluminum	Al	Selenium	Se
Silicon	Si	Bromine	Br
Phosphorous	P	Rubidium	Rb
Sulfur	S	Strontium	Sr
Chlorine	Cl	Yttrium	Y
Potassium	K	Zirconium	Zr
Calcium	Ca	Molybdenum	Mo
Titanium	Ti	Palladium	Pd
Vanadium	V	Silver	Ag
Chromium	Cr	Cadmium	Cd
Manganese	Mn	Indium	In
Iron	Fe	Tin	Sn
Cobalt	Co	Antimony	Sb
Cerium	Ce	Cesium	Cs
Nickel	Ni	Barium	Ba
Copper	Cu	Lanthanum	La
Zinc	Zn	Mercury	Hg
Gallium	Ga	Lead	Pb

<sup>1</sup> Analytical measurement determined by X-ray Fluorescence Analysis (XRF) EPA Method IO-3.3

## **A6.2 SITE LOCATION**

The selected monitoring sites for the PM<sub>2.5</sub> speciation monitoring are described in the various QAPPs approved by the EPA (Point Lay, version 1.4, Wainwright version 2.0, Deadhorse version 2.0, and Kaktovik version 1.1). Point Lay and Wainwright monitoring locations are expected to be representative of PM<sub>2.5</sub> chemical speciation concentrations observed in and near the Chukchi Sea. Deadhorse and Kaktovik monitoring locations are expected to be representative of the Beaufort Sea.

## **A6.3 WORK SCHEDULE/PROJECT TASKS**

Personnel working on the monitoring project will be selected by the SLR project manager and deemed fully qualified, trained, and capable of assigned tasks. Work schedules shall be determined by the SLR project manager and will include:

- Field or laboratory data review,
- Data quality assurance and quality control (QA/QC),
- Start-up calibrations,
- Field operation of samplers,
- Data summary reports (quarterly and annual),
- Preventive maintenance, and
- Corrective actions and reactive maintenance of unplanned system failures.

The monitoring period for the PM<sub>2.5</sub> speciation monitoring is anticipated to begin by January 17, 2012 but collection of PM<sub>2.5</sub> chemical speciation samples are expected to begin January 28, 2012 due to delays in sampler manufacturing. Sampling is expected to continue for a minimum of one year after initial commencement of exploration drilling which is scheduled to begin in July 2012. Monitoring beyond July 2013 will be determined by future air quality permit conditions. Table A-5 provides the work schedule for this monitoring project.



**Table A-5: Work Schedule for PM<sub>2.5</sub> Speciation Monitoring**

Monitoring Event	Date
QAPP Preparation, Equipment Procurement	December 2011 – January 2012
Submit QAPP for EPA approval	On or before January 17, 2012
Sampler installation and calibration <sup>1</sup>	January 25, 2012
First Sample Collected <sup>2</sup>	January 28, 2012
Quarterly and annual reports prepared and issued within 45 days of completion of the monitoring period <sup>3</sup>	Varies

<sup>1</sup> Monitoring is expected to occur at Deadhorse and Wainwright in 2012.

<sup>2</sup> Permits R10OCS/PSD-AK-09-01 and R10OCS/PSD-AK-2010-0 were issued and became effective January 27, 2012. The first scheduled sample collection day will be January 28, 2012.

<sup>3</sup> Although monitoring begins January 28, 2012, the monitoring period is based on a calendar quarter as required in the air quality permits. For purposes of calculating data completeness, sampling events that would have occurred prior to January 28 will not be counted as possible events for the quarter.

#### **Monitoring Year Quarters <sup>1</sup>**

Monitoring Quarter A	January 1, 2012 – March 31, 2012
Monitoring Quarter B	April 1, 2012 – June 30, 2012
Monitoring Quarter C	July 1, 2012 – September 30, 2012
Monitoring Quarter D	October 1, 2012 – December 31, 2013

<sup>1</sup> Schedule will be extended in similar fashion if monitoring is extended beyond July 2013.

## **A6.4 PROJECT RECORDS/REPORTS**

Table A-6 lists the project records and reports that will be included in the monitoring project. Project records and reports will include calibration documentation, quality control verification documentation, and annual monitoring data summary reports. If needed, corrective action reports and plans may be included in project documentation. Annual data summary reports will include supporting field and laboratory QC documentation.

Table A-6: Summary of Project Records and Reports

Records / Reports	Frequency	Content	Individual Responsible	Distribution
Calibration	Initial Setup	Accuracy Assessment	Field Operator	SLR Project Manager
Sampling Period QC Verifications	Before and after each monthly sampling period	Accuracy Assessment Summary	Field Operator	SLR Project Manager
Lab Data Summary	After each batch analysis	Analytical Data	Laboratory Project Manager	SLR Project Manager
Quarterly Summary Report	Quarterly	Summarize data	Data Manager	SLR Project Manager, SOI, EPA
Annual Summary Report	Annually	Summarize data	Data Manager	SLR Project Manager, SOI, EPA
Corrective Action Reports and responses	As Needed	Summarizes corrective actions taken to restore station to compliant status	SLR Project Manager	Field Operator, included in Annual Summary Report if needed

## A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT

Measurement Quality Objectives (MQOs) are designed to evaluate and control various phases (sampling, preparation, and analysis) of the measurement process to ensure that total measurement uncertainty is within the range prescribed by the data quality objectives (DQOs).

MQOs can be defined in terms of precision, accuracy, representativeness, detectability, completeness, and comparability.

Precision is an assessment of the mutual agreement among multiple independent measurements under similar conditions. EPA STN sampling does not require the operation of field precision samplers. Precision assessments are generally performed at the laboratory during the analytical process by repeated measurement of the same sample to assess analytical method reproducibility. Consistent with EPA STN sampling, precision will be assessed by methods specific to each analytical method. Table A-7 provides a summary of the methods used to determine analytical precision

**Table A-7: Methods for Determining Analytical Precision**

Analytical Method	Precision Method
Gravimetric / Mass	Standard Deviation of control filters
X-ray Fluorescence (XRF)	Replicate sample analysis
Ion Chromatography (IC)	Standard Deviation of QA standards
Thermal / Optical Reflectance or Transmission (TOR/TOT)	Replicate sample analysis

Accuracy is a measure of the proximity of a given measurements and the true value. Accuracy includes components of both random error and systematic error associated with instrumental bias. Accuracy will be assessed for field operations during sampler quality control flow calibration verifications. Additionally, during laboratory analysis, accuracy will be assessed by the analysis of samples containing known amounts of target species and elements.

Data completeness is a measure of the amount of data actually collected compared with the amount of data that could have been collected during the defined monitoring period, expressed as a percentage. Data completeness is calculated by dividing the number of valid samples by the total number of possible samples that were planned for collection during the monitoring period. The EPA STN QAPP does not clearly provide specific criteria or guidance for PM<sub>2.5</sub> speciation data completeness goals. The EPA STN QAPP adopts 40 CFR Part 50 data completeness requirements which only address priority pollutants. PM<sub>2.5</sub> mass is the only priority pollutant measured in the Beaufort and Chukchi Sea PM<sub>2.5</sub> speciation program. Both the STN and Interagency Monitoring of Protected Visual Environments (IMPROVE) indicate that a differentiation is made between recovery rate and data completeness. Recovery rate refers to the data completeness of the PM<sub>2.5</sub> mass measurement while completeness refers to the validity of the entire sample. Entire sample validity requires valid sample concentrations be obtained for PM<sub>2.5</sub>, sulfate, organic carbon, elemental carbon, and nitrate. Consistent with 40 CFR Part 50 priority pollutant data completeness requirements, the MQO goal for overall data completeness during the monitoring project is defined as 80 percent data capture for entire sample validity as defined by valid concentrations for PM<sub>2.5</sub> mass, sulfate, organic carbon, elemental carbon, and nitrate for an individual sample.

The MQOs for detection capabilities of the project are defined in terms of measurement resolution. Due to the complex nature of the sample collection and measurement procedures in the lab, species and element detection limits are not provided in this QAPP. However, refer to appendix D for descriptions of the analytical methods and expected project detection limits.

Detectability, method comparability, and representativeness are determined by the siting and exposure of the PM<sub>2.5</sub> speciation sampler and the use of approved analytical methods. The space-time domain and aspects for this specific application have been considered for each measurement parameter as prescribed in *Meteorological Monitoring Guidance for Regulatory Modeling Applications*, Section 3.1.1 Objective for Siting (EPA-454/R-99-005). PM<sub>2.5</sub> criteria were applied to assess the siting for this project. Additionally, the selected analytical laboratory and methodology selected for the project is consistent and comparable to those used for the EPA STN.

A number of laboratory quality control procedures are employed to assess the accuracy and precision of analytical procedures. In some instances, these procedures or acceptance criteria are more stringent than those described in the IMPROVE or CSN / STN monitoring network.

XRF analysis utilizes multi-elemental quality control standard to assess method precision. Six (6) elements are selected as critical quality control indicators and measurement control for XRF analysis is based on their performance.  $\pm 10\%$  error (%E) is used as the project acceptance criteria for this measurement. Figure A-3 provides a sample quality control report for the multi-elemental quality control sample.

Additional XRF accuracy assessments are made using NIST Standard Reference Materials (SRM). These materials provide a more stringent acceptance criteria than specified in the IMPROVE CSN/STN monitoring network. These criteria will also be used to assess the measurement accuracy for XRF analyses. Figure A-4 provides a sample quality control report for the NIST SRM sample.

Sample replicate analysis for XRF analyses is also performed and relative percent difference (RPD) will be used to assess analysis precision. RPD calculations will only be performed if analytical measurement indicates concentrations at least three times the level of the measurement uncertainty. Due to the expected low concentrations of PM<sub>2.5</sub> mass, and associated low concentrations of chemical constituents, failure to achieve the specified project precision goal will not be considered a critical project failure but will be discussed in the data summary report.

## Micromatter Multi-elemental Quality Control Standard: QS285

## QC Standard Results

Analyte	n	Counts per Second			c.v.	%E
		Calib.	Meas.	S.D.		
Si(0)	13	185.90	179.67	2.92	1.63	-3.35
Si(1)	13	11.44	11.45	0.19	1.62	0.15
Ti(2)	13	132.64	134.36	1.38	1.03	1.29
Fe(3)	13	179.51	178.77	2.36	1.32	-0.41
Se(4)	12	42.27	41.58	0.88	2.11	-1.63
Pb(4)	12	30.54	30.63	0.86	2.81	0.29
Cd(5)	13	42.52	42.60	0.64	1.50	0.17

Figure A-3: Sample XRF Multi-elemental Quality Control Report.

## NIST Standard Reference Materials: SRM 1832, SRM 1833

Analyte/ SRM	n	Certified Value(μg/cm²)	Measured Value (μg/cm²)				% Rec.
			High	Low	Average		
Al 1832	45	14.6 +/- .97	15.90	13.86	14.44	+/- 0.46	98.9
Si 1832	45	34.0 +/- 1.1	38.01	33.37	34.59	+/- 1.04	101.7
Si 1833	45	33.0 +/- 2.1	33.16	30.67	32.25	+/- 0.49	97.7
S 2708	45	2.46 +/- .25	2.57	2.28	2.44	+/- 0.08	99.0
K 1833	45	17.3 +/- 1.64	17.44	16.09	16.91	+/- 0.34	97.7
Ca 1832	45	19.4 +/- 1.30	21.35	19.95	20.74	+/- 0.34	106.9
Ti 1833	45	12.8 +/- 1.79	12.85	11.72	12.28	+/- 0.37	95.9
V 1832	45	4.70 +/- .49	4.95	4.37	4.64	+/- 0.17	98.6
Mn 1832	45	4.54 +/- .49	4.84	4.56	4.69	+/- 0.06	103.3
Fe 1833	45	14.2 +/- .45	14.49	13.97	14.21	+/- 0.13	100.1
Cu 1832	45	2.43 +/- .16	2.49	2.31	2.43	+/- 0.04	99.8
Zn 1833	45	4.01 +/- .23	4.07	3.82	3.95	+/- 0.05	98.4
Pb 1833	45	16.7 +/- .85	18.26	15.97	16.94	+/- 0.46	101.5

Figure A-4: Sample XRF Standard Reference Material Quality Control Report.

Ion chromatography (IC) analysis utilizes laboratory control samples (LCS) and matrix spikes to assess measurement accuracy. Matrix spike samples are prepared by adding a known amount of analyte concentration to an actual sample and measuring the recovery of that known amount to assess sample media interferences. LCS and matrix spike samples are considered critical measurement quality control samples and  $\pm 10\%$  acceptance criteria is used as acceptance criteria for this measurement. Figure A-5 provides a sample quality control report for the IC quality control samples.



IC analysis also utilizes laboratory blank samples to assess any sort of contamination that may have been introduced during the sample preparation and analysis procedures. Blank results are not considered critical measurement quality control samples however samples batches where the blanks contain measured analyte concentrations above the instrument detection limits will be re-analyzed and the source of the contamination will be identified. Figure A-6 provides a sample blank quality control report for the IC quality control samples.

IC analysis also utilizes initial calibration verification (ICV) and continuing calibration verification (CCV) quality control samples. Low level and mid level standards are used to assess measurement accuracy across the range of expected sample concentrations. ICV and CCV samples are considered critical measurement quality control samples and acceptance criteria of  $\pm 10\%$  are used as acceptance criteria for this measurement. Figure A-7 provides a sample quality control report for the IC calibration quality control samples.

Analyte	Sample ID	Sample Conc. $\mu\text{g/mL}$	Spike Conc. $\mu\text{g/mL}$	Spike Amount $\mu\text{g/mL}$	Percent Recovery
Cl	LCS	0.074	9.29	10.0	92.2
Cl	10-T5537	5.04	14.8	10.0	97.7
SO <sub>4</sub>	LCS	< 0.05	9.50	10.0	95.0
SO <sub>4</sub>	10-T5537	3.71	13.3	10.0	95.8
NH <sub>4</sub>	LCS	< 0.05	10.8	10.0	108.
NH <sub>4</sub>	10-T5539	1.54	6.73	5.00	104.

**Figure A-5: Sample IC Laboratory Control Sample Quality Control Report.**

Analyte	Sample ID	Measured Conc. $\mu\text{g/mL}$	MDL Conc. $\mu\text{g/mL}$
Cl	ICB	< MDL	0.050
Cl	Prep_Blk	< MDL	0.050
Cl	Meth_Blk*	0.740	0.500
Cl	CCB	< MDL	0.050
SO <sub>4</sub>	ICB	< MDL	0.050
SO <sub>4</sub>	Prep_Blk	0.051	0.050
SO <sub>4</sub>	Meth_Blk*	< MDL	0.500
SO <sub>4</sub>	CCB	< MDL	0.050
NH <sub>4</sub>	ICB	< MDL	0.050
NH <sub>4</sub>	Prep_Blk	< MDL	0.050
NH <sub>4</sub>	Meth_Blk*	< MDL	0.500
NH <sub>4</sub>	CCB	< MDL	0.050

**Figure A-6: Sample IC Blank Quality Control Report.**

Analyte	Sample ID	Standard Conc. $\mu\text{g/mL}$	Measured Conc. $\mu\text{g/mL}$	Percent Recovery
Cl	ICV_LO	1.00	0.98	98.3
Cl	ICV_MID	10.0	9.72	97.2
Cl	CCV_LO	1.00	0.96	96.5
Cl	CCV_MID	10.0	9.61	96.1
SO4	ICV_LO	1.00	1.07	106.6
SO4	ICV_MID	10.0	9.97	99.7
SO4	CCV_LO	1.00	1.00	100.3
SO4	CCV_MID	10.0	9.84	98.4
NH4	ICV_LO	0.500	0.55	109.6
NH4	ICV_MID	5.00	5.25	105.0
NH4	CCV_LO	0.500	0.55	110.0
NH4	CCV_MID	5.00	5.24	104.8

Figure A-7: Sample IC Calibration Quality Control Report.

Organic and elemental carbon (TOR/TOT) analysis utilizes a sucrose laboratory control sample (LCS) to assess measurement accuracy. LCS samples are considered critical measurement quality control samples and a  $\pm 10\%$  acceptance criterion is used as acceptance criteria for this measurement. Figure A-8 provides a sample quality control report for the TOR/TOT quality control samples.

Laboratory blank samples are used in TOR/TOT analysis to assess contamination that may have been introduced during the sample preparation and analysis procedures. Blank results are not considered critical measurement quality control samples however samples batches where the blanks contain measured analyte concentrations above the instrument detection limits will be re-analyzed and the source of the contamination will be identified. Figure A-9 provides a sample blank quality control report for the TOR/TOT quality control samples.

*Sucrose Standard*

Sample ID	Sucrose Std. Conc. $\mu\text{g/cm}^2$	Measured Conc. $\mu\text{g/cm}^2$	Percent Recovery
Sucrose 10ul 11/11/2011	28.06	27.94	99.6
Sucrose 10ul 11/14/2011	28.06	28.61	102.0

Figure A-8: Sample OC/EC Calibration Quality Control Report

Blank Data

Sample ID	Analyte	Measured Conc. $\mu\text{g/cm}^2$	MDL $\mu\text{g/cm}^2$
11/11/2011	OC	<MDL	0.2
	EC	<MDL	0.2
11/14/2011	OC	<MDL	0.2
	EC	<MDL	0.2

Figure A-9: Sample OC/EC Blank Quality Control Report.

**Table A-8: Summary of MQOs for the PM<sub>2.5</sub> Chemical Speciation Monitoring Program**

Quality Objective	Assessment Method	Acceptance Criteria <sup>1</sup>	Corrective Actions
Accuracy	Sampler Flow Verifications	± 10%	Re-calibrate sampler flow
	Laboratory Quality Control Samples	Gravimetric / Mass - ± 5 µg X-ray Fluorescence (XRF) / Elements - ± 10% Ion Chromatography (IC) / Ions - ± 10% Elemental / Total Carbon (TOR / TOT) - ± 10%	Re-calibrate, re-analyze QC and field samples
Precision <sup>2</sup>	Laboratory Replicates	Gravimetric / Mass - ± 5 µg X-ray Fluorescence (XRF) / Elements - ± 7% or 5% Ion Chromatography (IC) / Ions - ± 10% Elemental Carbon (TOR / TOT) - ± 10% Total Carbon (TOR / TOT) - ± 10%	Re-calibrate, re-analyze QC and field samples
Data Completeness	Calculated	≥ 80% per monitoring quarter (valid samples defined by combined PM <sub>2.5</sub> , Sulfate, OC, EC, Nitrate valid measurements for the sampling event)	Collect additional samples; Re-analysis of samples by laboratory
Representativeness	Sampler siting	Meets PM <sub>2.5</sub> requirements	N/A
Detectability <sup>3</sup>	Laboratory determined detection limits	Gravimetric / Mass - 0.3 µg/m <sup>3</sup> X-ray Fluorescence (XRF) / Elements - 0.05 - 20 ng/m <sup>3</sup> Ion Chromatography (IC) / Ions - 10 - 100 ng/m <sup>3</sup> Elemental Carbon (TOR / TOT) - 100 ng/m <sup>3</sup> Total Carbon (TOR / TOT) - 250 ng/m <sup>3</sup>	N/A
Comparability	Analytical methodology	Analytical laboratory experienced with STN/IMPROVE Measurement methods consistent with STN/IMPROVE	N/A

<sup>1</sup> Laboratory acceptance criteria defined from IMPROVE MQO goals. *Interagency Monitoring of Protected Visual Environments, Quality Assurance Plan OAQPS Category 1 QAPP*. Revision 0.0 March 2002. Section 4.6 Measurement Quality Objectives.

<sup>2</sup> Precision objectives for measurements that are significantly above instrument detection limits. Assessment of measurements near or below detection limits exaggerates statistical uncertainty. XRF precision criteria depends upon the number of fluorescers for a particular element.

<sup>3</sup> Detection limits vary for each analytical parameter.



## **A8 SPECIAL TRAINING/CERTIFICATIONS**

Field personnel working on this project will have their qualifications reviewed and will be determined by the project manager to have sufficient experience and knowledge in PM<sub>2.5</sub> monitoring. Laboratory personnel will have their qualifications reviewed by the laboratory manager and will be determined to have sufficient experience and knowledge in analytical procedures.

All SLR field personnel will be trained in site-specific safety and logistics. Training will be conducted by the SLR project manager or his designees. Site-specific training checklists will document the specific training received by each field technician (Appendix C). Training in laboratory procedures will be provided by the laboratory manager. Sample laboratory procedures are provided in Appendix D.

Training on the proper setup, calibration, and operation of the PM<sub>2.5</sub> chemical speciation samplers will be provided by completing the training videos provided on the EPA Technology Transfer Network Ambient Monitoring Technology Information Center (AMTIC). Training on field standard operating procedures and sampler setup for chemical speciation samplers is provided at <http://www.epa.gov/ttnamti1/spectraining.html>. Content provided in these training materials will be supplemented by the manufacturer-supplied operating manual and directly through discussions with the manufacturer technical support staff.

## **A9 DOCUMENTS AND RECORDS**

Table A-9 provides a list of the documents and records that will be generated as part of the project and included in the applicable data reports. Copies of all field documentation will be maintained on-site and at the SLR office. All electronic data will be stored on a server at the SLR office and will be backed up periodically to alternate storage locations to prevent data loss. Laboratory records will be stored at the laboratory facility and backed up according to laboratory data management procedures.

Sample project forms are included in Appendix C. Data report submittal and content requirements are provided in section C2.

**Table A-9: Summary of Documents and Records**

<b>Document Type</b>	<b>Frequency</b>	<b>Preparer</b>
Calibration Sheets	Monthly	Station Operator
Maintenance Log	As needed	Station Operator
Sample Chain of Custody Forms	Each batch shipment	Station Operator
Laboratory Analytical Summary Reports	Each filter batch	Laboratory Manager
Quarterly Data Report	Quarterly	Data Manager
Annual Data Report	Annually	Data Manager
QAPP Revisions	As needed	Project Manager

## B. MEASUREMENT AND DATA ACQUISITION

### B1 SAMPLING PROCESS DESIGN

The primary objective of the PM<sub>2.5</sub> Speciation Monitoring Program is to collect PM<sub>2.5</sub> chemical speciation data and comply with post construction monitoring requirements specified in permit number R10OCS/PSD-AK-09-01 (Noble Discoverer Drillship - Chukchi Sea Exploration Drilling Program) and R10OCS/PSD-AK-2010-01 (Noble Discoverer Drillship - Beaufort Sea Exploration Drilling Program).

To accomplish this objective, PM<sub>2.5</sub> chemical speciation data, including sulfates, nitrates, organics, metals, sea salt and crustal matter will be collected at a location representative of the Beaufort and Chukchi Seas. Point Lay and Wainwright monitoring locations are expected to be representative of concentrations observed in and near the Chukchi Sea. Deadhorse and Kaktovik monitoring locations are expected to be representative of the Beaufort Sea.

Chukchi Permit (R10OCS/PSD-AK-09-01) condition R.1.3 and Beaufort Permit (R10OCS/PSD-AK-2010-01) condition S.1.3 specify the monitoring period shall commence within 120 days after the final permit is issued and continue for a minimum of 1 year after commencement of initial operation of the Discoverer as an OCS Source. This suggests a sampling program from January, 2012 until at least July, 2013.

EPA STN samples are collected on a once every three day or once every six day schedule all year-round to assess national trends in background pollutant concentrations. Higher frequency sampling schedule is generally followed by locations where PM<sub>2.5</sub> concentrations tend to be relatively high or pose a health concern. Because observed PM<sub>2.5</sub> mass concentrations along the Chukchi and Beaufort Seas have historically been very low, the project adopts a one sample every six day frequency and schedule. The first scheduled sampling day is January 28, 2012. The sampling schedule is provided in Appendix B.

PM<sub>2.5</sub> chemical speciation samplers will supplement existing ambient air quality and meteorological monitoring equipment already in operation at the representative location. Met One SuperSASS™ samplers capable of collecting samples on multiple sample media (Teflon®, Nylasorb®, and quartz) will be used to ensure proper sample collection and handling. Met One SuperSASS™ samplers were selected after careful consideration of site characteristics and after consideration of site operator survey results provided in *Evaluation of PM<sub>2.5</sub> Chemical Speciation Samplers for Use in the EPA National PM<sub>2.5</sub> Chemical Speciation Network*, Table II-15 (EPA-454-R-01-005). Critical sampler characteristics include single person field installation ability, quick installation time, sampler ease of use for both sample collection and audit and calibration, and sampler operation reliability. SuperSASS™ samplers are currently used in the EPA STN sampling network in combination with another sampler specific to carbon sample collection.

EPA STN samples are collected and analyzed by EPA's contract laboratory in North Carolina. Since the EPA STN program and contract lab are geared toward state and local agency participation, the laboratory generally does not solicit work from other entities. Instead, an alternate, non-EPA contract lab (Chester LabNet) on the west coast will be used. This lab has experience performing PM<sub>2.5</sub> speciation analysis and is located on the west coast making sample shipment more convenient and reliable. The lab will use similar laboratory testing methodology as that used by the EPA STN contract laboratory. Tables A-3 and A-4 provide a list of the project target constituents and analytical procedures used for each.

Figure B-1 provides a map depicting the general project area. The Beaufort and Chukchi Seas combined represent approximately 1,000 miles of north Alaska coastline. Ambient air quality and meteorological monitoring is already taking place at Wainwright, Point Lay, Deadhorse, and Kaktovik communities. These monitoring stations will be expanded to include PM<sub>2.5</sub> speciation monitoring as described in this QAPP.

The Point Lay (A on Figure B-1) ambient air and meteorological monitoring site is located at the northeast corner (off of Tuttunniagvik Street just north of Sisrik Avenue) of the Point Lay Village, Alaska. The community of Point Lay is located on the Chukchi Sea coast approximately 180 miles southwest of Barrow and 2 miles southwest of the Kokolik River. Figure B-2 provides a view of the Point Lay station. For more specifics regarding the Point Lay monitoring program, refer to the approved Point Lay ambient air and meteorological monitoring QAPP.

The Wainwright (B on Figure B-1) ambient air quality and meteorological monitoring site is located on the east side of the community of Wainwright. The community of Wainwright is located on the Chukchi Sea coast approximately 90 miles southwest of Barrow and 3 miles northeast of the Kuk River estuary (also known as the Wainwright Inlet or Lagoon). The monitoring site is located on a broad flat coastal plain that is dotted with several small lakes and ponds at an elevation of approximately 26 feet above sea level. Figure B-3 provides a view of the Wainwright station. For more specifics regarding the Wainwright monitoring program, refer to the approved Wainwright ambient air and meteorological monitoring QAPP.

The Deadhorse (C on Figure B-1) ambient air monitoring site is located in the town of Deadhorse, Alaska along the arctic coastal plain approximately 12 kilometers (km) south of the shoreline and approximately 160 km north of the Brooks Range. The station is located approximately 61 feet above sea level and is surrounded by light industrial facilities within the town of Deadhorse. Figure B-4 provides a view of the Deadhorse station. For more specifics regarding the Deadhorse monitoring program, refer to the approved Deadhorse ambient air and meteorological monitoring QAPP.

The Kaktovik (D on Figure B-1) ambient air and meteorological monitoring site is located along the landfill access road southeast of the Kaktovik Village on Barter Island on the Beaufort Sea. The station is located at approximately 35 feet above sea level and is surrounded by tundra vegetation consisting of low shrubs, sedges, and mosses. Figure B-5 provides a view of the Kaktovik station. For more specifics regarding the Kaktovik monitoring program, refer to the approved Kaktovik ambient air and meteorological monitoring QAPP.



**Figure B-1: PM<sub>2.5</sub> Speciation Monitoring Program Site Map**





**Figure B-2: Point Lay Monitoring Station**



**Figure B-3: Wainwright Monitoring Station**





**Figure B-4: Deadhorse Monitoring Station**





\* - Tower is no longer attached to the shelter. Updated photo is not available at the time of QAPP submission.

**Figure B-5: Kaktovik Monitoring Station**

## **B2 SAMPLING METHODS**

Met One Spiral Aerosol Speciation Samplers SuperSASS™ PM<sub>2.5</sub> Speciation samplers will be used. This sampler provides multiple parallel sample cassettes. Each cassette has its own cyclone size-selective PM<sub>2.5</sub> inlet, denuder (if applicable), and tandem filter holder. The cassettes are mounted in an aspirated solar shield that maintains the eight independent sampling cassettes at the same temperature within 3°C during and after a sample event. The cassette inlets point downward. Each channel is independently flow-controlled to 6.7 liters per minute (LPM) and volumetric flow and volume are logged each five minutes. Ambient barometric pressure and temperature are also measured and logged. The sampler incorporates a PM<sub>2.5</sub> Sharp Cut Cyclone aerosol separator which requires no grease or oil. The PM<sub>2.5</sub> cyclone inlet is designed to be removable from the cassette which allows either field or laboratory maintenance. The filter samples are transported to the laboratory for analysis in their filter cassettes. Sample recovery and cleaning in the laboratory insures the inlet and sampling cassette is free from contamination each sample event. The sampler is designed without a plenum or sample tube which requires frequent field maintenance. The Fine Particle sample exits the cyclone and is immediately captured on the filter cassette. The sample cassettes are designed to use one or two stacked 46.2-mm filters. Any cassette can be configured with one or two filters or a denuder followed by one or two filters. Figures B-6 and B-7 provide views of sampler schematic.

Monitoring equipment will be operated using Prevention of Significant Deterioration (PSD) quality assurance practices as a guide. In general, samplers will be calibrated and operated according to manufacturer recommendations and consistent with EPA STN quality assurance procedures. Calibrations and periodic flow-rate verifications will be documented and provided as quality assurance documentation for the monitoring. Independent audits will be performed on the samplers. Collocated sampler operation for PM<sub>2.5</sub> speciation monitoring is not planned. Section B5 describes the project Quality Control procedures.

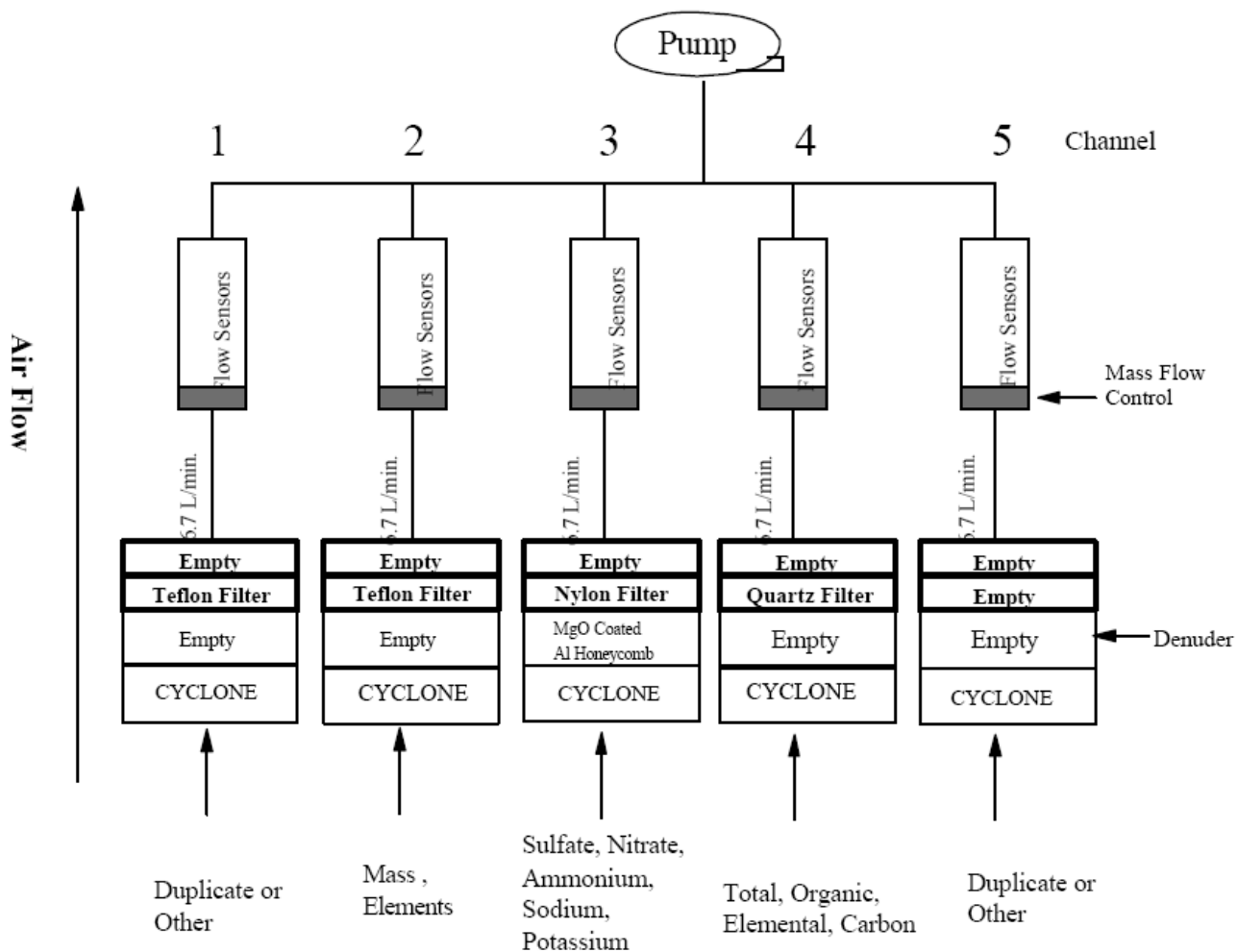
Section B4 provides a description of the different sampling media used for the project. The sampling schedule is provided in Appendix B. Sampling is expected to occur on a once every six day schedule and sample collection time is expected to be for 24 hours.<sup>1</sup>

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<sup>1</sup> 40 CFR Part 50, Appendix L indicates that a sampling period of 1,380 to 1,500 minutes (23 hours to 25 hours) for PM<sub>2.5</sub> is acceptable.



**Figure B-6: Met One Spiral Aerosol Speciation Sampler (SASS™)**



**Figure B-7: Schematic of SASS Sampling System**

### **B3      SAMPLE HANDLING AND CUSTODY**

An alternate sample handling and custody procedure will be used than what is typically used for EPA STN/IMPROVE sampling. EPA STN sites require samples to be shipped to the EPA contract laboratory within 48 hours of sample collection. Given the logistics involved in shipping samples out of Alaska monitoring locations, an alternate shipping schedule will be used. Samples will be retrieved within 48 hours of collection but then held in cold storage (<4 °C) and shipped to the laboratory in batches. Sample handling will adhere to PM<sub>2.5</sub> mass holding time guidelines. Sample collection will occur within 30 days of initial filter preparation at the lab. Laboratory analysis will occur within 30 days of sample collection in the field. Sample transport and receipt will be tracked using standard chain of custody forms.

Due to the samples being collected on filters, samples originate in the laboratory, transfer to the field for exposure, then return to the laboratory for analysis. Figure B-8 provides a summary of the filter handling process for the project.

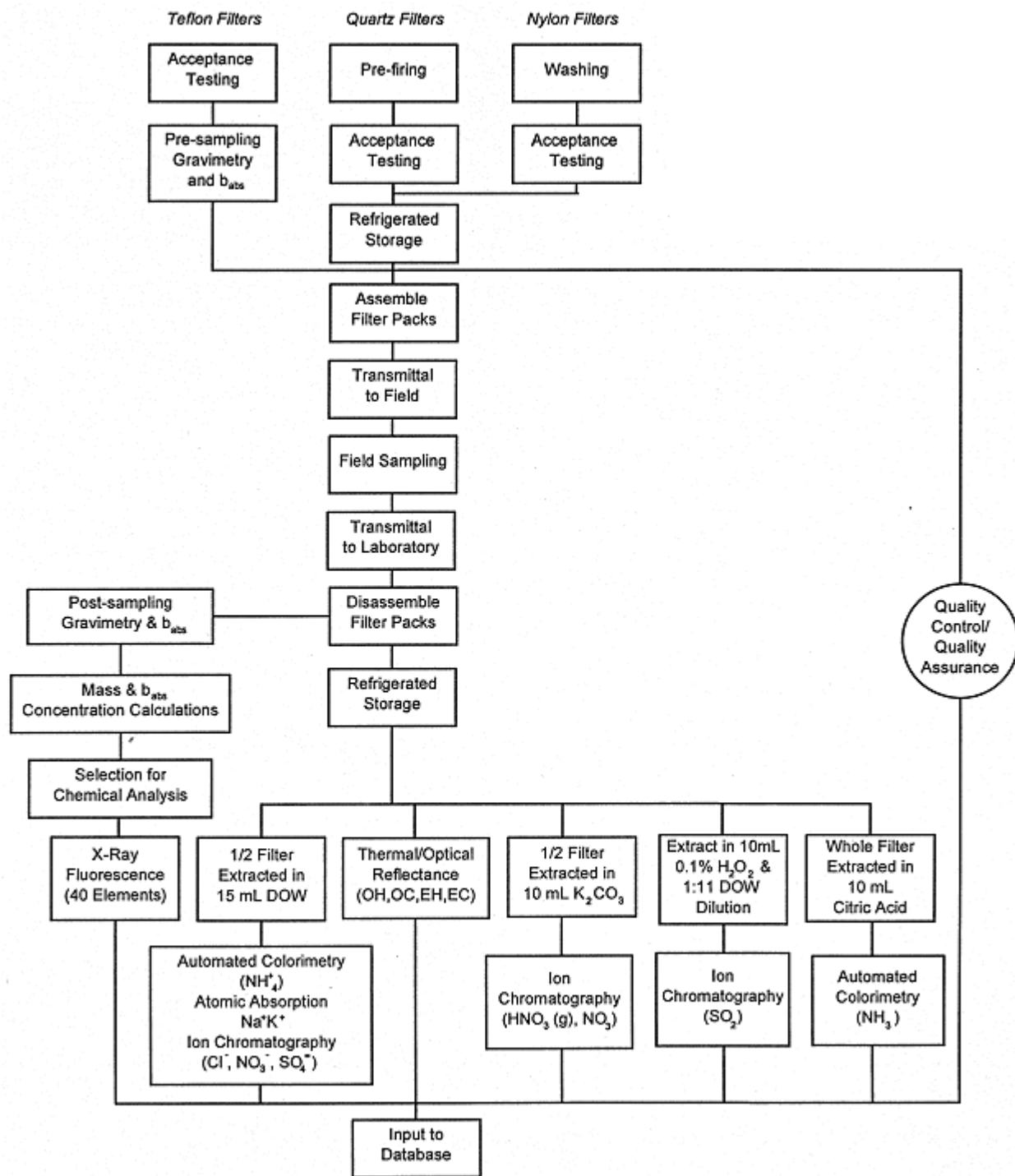


Figure B-8: Filter Flow Diagram Acceptance through Analysis



## **B4 ANALYTICAL METHODS REQUIREMENTS**

### **B4.1 MASS MEASUREMENT METHODS**

Particulate mass concentration is determined by gravimetric analysis using a Teflon® filter media. Net mass on a filter is determined by weighing the filter before and after sampling with a balance in a temperature- and relative humidity-controlled environment. To minimize particle volatilization and aerosol liquid water bias, PM<sub>2.5</sub> reference methods require that filters be equilibrated for 24 hours at a constant (within  $\pm 5\%$ ) relative humidity between 30% and 40% and at a constant (within  $\pm 2^\circ\text{C}$ ) temperature between 20°C and 23°C.

Balance calibrations are established before and after each weighing session using Class M and Class S standards traceable to National Institute of Standards and Technology (NIST) mass standards. Periodic verifications take place every ten filters processed during weighing sessions. Approximately one out of ten filters are re-weighed and used to calculate the precision of the measurement. When quality control results differ from specifications by more than 5% the samples are re-analyzed.

### **B4.2 ELEMENTAL ANALYSIS METHODS**

Elemental analysis is performed using X-ray fluorescence (XRF). XRF is the most commonly used elemental analysis method due to the multi-element capabilities, relatively low cost, low detection limits, and preservation of the filter for other analyses.

In XRF, the filter deposit is irradiated by high energy x-rays (XRF) or protons (PIXE) which eject inner shell electrons from the atoms of each element in the sample. When a higher energy electron drops into the vacant lower energy orbital, a fluorescent x-ray photon is released. The energy of this photon is unique to each element, and the number of photons is proportional to the concentration of the element. Concentrations are quantified by comparing photon counts for a sample with those obtained from thin-film standards of known concentration. XRF analysis is usually performed on Teflon-membrane filters.

Quality control standards and replicates from previous batches are analyzed for every 10 to 20 samples. When quality control results differ from specifications by more than  $\pm 10\%$ , or if the replicate concentrations differ from the original values (assuming they are at least 10 times detection limits) by more than  $\pm 10\%$ , the samples are re-analyzed.

### **B4.3 ION CHROMATOGRAPHY METHODS**

Ion chromatography (IC) methods rely on the fact that the target analyte is soluble in water. All IC analysis methods require a fraction of the filter to be extracted in water and then filtered to remove insoluble residues prior to analysis. The sample extract passes through an ion-exchange column which separates the ions in time for individual quantification, usually by an electro-conductivity detector. Quartz or Nylon (Nylasorb®) filter media are used to collect samples for IC. Nylon filters are the preferred media for nitrate analysis due to the ability to better retain volatile nitrate constituents. Quartz media will be used as a secondary, back-up sampling media to maximize data completeness in the event that nylon filters are damaged or unable to be collected.

Calibration standards are prepared weekly or biweekly in routine operation of ionic analysis. New calibration standards are verified with the previous week's calibration standards to ensure consistency among the measurements. During instrument calibration, if any calibration point varies by more than  $\pm 10\%$  of the specified value, the system is rechecked or a new calibration standard prepared to ensure the accuracy of the analysis.

### **B4.4 CARBON ANALYSIS METHODS**

Three classes of carbon are commonly measured in ambient aerosol samples collected on quartz-fiber filters: (1) organic, volatilized, or non-light absorbing carbon; (2) elemental or light-absorbing carbon; and (3) carbonate carbon.

Thermal/optical reflectance (TOR) and Thermal/optical transmittance (TOT) methods of carbon analysis utilizes volatilization at high temperatures to quantify carbon. Temperatures ranging from ambient to 550 °C to 800 °C are used to volatilize carbon, convert it to methane and quantify it, usually with a flame ionization detector. Varying temperature ranges correspond to the different classifications of carbon.

Quality control standards and replicates are analyzed for every 10 to 20 samples. When quality control results differ from specifications by more than  $\pm 10\%$ , or if the replicate concentrations differ from the original values (assuming they are at least 10 times detection limits) by more than  $\pm 10\%$ , the samples are re-analyzed.

Table B-1: PM<sub>2.5</sub> Speciation Methods

AQS Code	Parameter	AQS Method Code	Sample Media	Sample Analysis Description	Lab Prep Method / SOP	Lab Analysis Method / SOP
88102	Antimony	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88103	Arsenic	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88104	Aluminum	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88107	Barium	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88109	Bromine	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88110	Cadmium	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88111	Calcium	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88112	Chromium	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88113	Cobalt	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88114	Copper	811	Teflon <sup>®</sup>	Energy dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88115	Chlorine	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88117	Cerium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88118	Cesium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88124	Gallium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88126	Iron	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88128	Lead	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88131	Indium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88132	Manganese	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88136	Nickel	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88140	Magnesium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3

Table B-1: PM<sub>2.5</sub> Speciation Methods (Continued)

AQS Code	Parameter	AQS Method Code	Sample Media	Sample Analysis Description	Lab Prep Method / SOP	Lab Analysis Method / SOP
88152	Phosphorus	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88154	Selenium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88160	Tin	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88161	Titanium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88164	Vanadium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88165	Silicon	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88166	Silver	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88167	Zinc	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88168	Strontium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88169	Sulfur	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88176	Rubidium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88180	Potassium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88184	Sodium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88185	Zirconium	811	Teflon <sup>®</sup>	Energy Dispersive XRF	N/A	XR-002.04 / EPA IO 3.3
88301	Ammonium Ion	812	Nylon	Ion Chromatography	IC-003.04	IC-009.01 / EPA 300.0 / 300.7
88302	Sodium Ion	812	Nylon	Ion Chromatography	IC-003.04	IC-009.01 / EPA 300.0 / 300.7
88303	Potassium Ion	812	Nylon	Ion Chromatography	IC-003.04	IC-009.01 / EPA 300.0 / 300.7
88304	Ocx Carbon	813	Quartz	Thermal Optical Transmittance	N/A	OC-001.03 / IMPROVE-A
88305	Oc Csn Unadjusted Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88306	Total Nitrate	812	Nylon	Ion Chromatography	IC-003.04	IC-009.01 / EPA 300.0 / 300.7
88307	Ec Csn Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A

Table B-1: PM<sub>2.5</sub> Speciation Methods (Continued)

AQS Code	Parameter	AQS Method Code	Sample Media	Sample Analysis Description	Lab Prep Method / SOP	Lab Analysis Method / SOP
88312	Total Carbon	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88320	Oc Tor	815	Quartz	OC1+OC2+OC3+OC4+OP	N/A	OC-001.03 / IMPROVE-A
88321	Ec Tor	815	Quartz	EC1+EC2+EC3-OP	N/A	OC-001.03 / IMPROVE-A
88324	Oc1	814	Quartz	IMPROVE TOR w/ Adjustment	N/A	OC-001.03 / IMPROVE-A
88325	Oc2	814	Quartz	IMPROVE TOR w/ Adjustment	N/A	OC-001.03 / IMPROVE-A
88326	Oc3	814	Quartz	IMPROVE TOR w/ Adjustment	N/A	OC-001.03 / IMPROVE-A
88327	Oc4	814	Quartz	IMPROVE TOR w/ Adjustment	N/A	OC-001.03 / IMPROVE-A
88328	Op Tor	814	Quartz	IMPROVE TOR w/ Adjustment	N/A	OC-001.03 / IMPROVE-A
88329	Ec1	814	Quartz	IMPROVE TOR	N/A	OC-001.03 / IMPROVE-A
88330	Ec2	814	Quartz	IMPROVE TOR	N/A	OC-001.03 / IMPROVE-A
88331	Ec3	814	Quartz	IMPROVE TOR	N/A	OC-001.03 / IMPROVE-A
88332	Oc1 Csn Unadjusted Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88333	Oc2 Csn Unadjusted Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88334	Oc3 Csn Unadjusted Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88335	Oc4 Csn Unadjusted Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88336	Op Csn Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88379	Op Tot	813	Quartz	STN TOT	N/A	OC-001.03 / IMPROVE-A
88385	Ec3 Csn_Rev Unadjusted	816	Quartz	IMPROVE A	N/A	OC-001.03 / IMPROVE-A
88403	Sulfate	812	Nylon	Ion Chromatography	IC-003.04	IC-009.01 / EPA 300.0 / 300.7
88502	PM <sub>2.5</sub> Speciation Mass	810	Teflon®	Gravimetric	N/A	GR-019.02

**Table B-2: PM<sub>2.5</sub> Speciation Method Detection Limits**

Parameter	Fed MDL <sup>1</sup>	Lab MDL µg/m3 <sup>2</sup>
Antimony	0.01476	0.009548
Arsenic	0.00247	0.001497
Aluminum	0.01088	0.007019
Barium	0.05876	0.073287
Bromine	0.00199	0.001135
Cadmium	0.0105	0.006813
Calcium	0.00347	0.001755
Chromium	0.00159	0.000877
Cobalt	0.00141	0.000723
Copper	0.00135	0.000723
Chlorine	0.00578	0.004232
Cerium	0.08603	*
Cesium	0.03689	0.029211
Gallium	0.00331	0.001858
Iron	0.00196	0.001135
Lead	0.00549	0.003458
Indium	0.01128	0.00929
Manganese	0.00231	0.001342
Nickel	0.00125	0.000723
Magnesium	0.01841	0.015741
Phosphorus	0.00627	0.00418
Selenium	0.00212	0.001239
Tin	0.01787	0.011922
Titanium	0.00208	0.001239
Vanadium	0.0015	0.000877
Silicon	0.00753	0.004903
Silver	0.01048	0.006658
Zinc	0.00145	0.000877

\* Lab MDL not available at the time of QAPP preparation.

<sup>1</sup> Fed MDL published in AQS on-line Codes and Descriptions guidance document.

<sup>2</sup> Detection limit estimated at 3 times the standard deviation of the measurement background. Uncertainty factor of 2 included. MDL estimated based on 9.88 m<sup>3</sup> expected volume of ambient air drawn through the filter. Concentrations below this limit will be reported but noted as being below this level. Sample-specific detection limits may vary based on volume of air sampled and potential analytical interferences.



**Table B-2: PM<sub>2.5</sub> Speciation Method Detection Limits (Continued)**

Parameter	Fed MDL <sup>1</sup>	Lab MDL µg/m <sup>3</sup> <sup>2</sup>
Strontium	0.00251	0.001961
Sulfur	0.00662	0.003458
Rubidium	0.00217	0.001239
Potassium	0.00341	0.002632
Sodium	0.05107	0.13109
Zirconium	0.00359	0.002219
Ammonium Ion	0.017	0.025805
Sodium Ion	0.03	0.05161
Potassium Ion	0.014	0.025805
Ocx Carbon	0.134	0.010322
Oc Csn Unadjusted Tot	0.245	0.010322
Total Nitrate	0.008	0.025805
Ec Csn Tot	0.245	0.010322
Total Carbon	0.245	0.010322
Oc Tor	1.005	0.144509
Ec Tor	0.233	0.144509
Oc1	0.11	0.010322
Oc2	0.208	0.010322
Oc3	0.686	0.010322
Oc4	0.27	0.010322
Op Tor	0.196	0.010322
Ec1	0.147	0.010322
Ec2	0.184	0.010322
Ec3	0.061	0.010322
Oc1 Csn Unadjusted Tot	0.245	0.010322
Oc2 Csn Unadjusted Tot	0.245	0.010322
Oc3 Csn Unadjusted Tot	0.245	0.010322
Oc4 Csn Unadjusted Tot	0.245	0.010322
Op Csn Tot	0.245	0.010322
Op Tot	0.245	0.010322
Ec3 Csn_Rev Unadjusted	0.03	0.010322
Sulfate	0.012	0.025805
PM2.5 Speciation Mass	0.104	0.05161

<sup>1</sup> Fed MDL published in AQS on-line Codes and Descriptions guidance document.

<sup>2</sup> Detection limit estimated at 3 times the standard deviation of the measurement background. Uncertainty factor of 2 included. MDL estimated based on 9.88 m<sup>3</sup> expected volume of ambient air drawn through the filter. Concentrations below this limit will be reported but noted as being below this level. Sample-specific detection limits may vary based on volume of air sampled and potential analytical interferences.

## **B5      QUALITY CONTROL**

Quality Assurance/Quality Control (QA/QC) procedures are required for this project to ensure that the collected data meet standards of reliability and accuracy.

The PM<sub>2.5</sub> speciation samplers will be installed and calibrated according to manufacturer procedures. Flow quality control verifications will be performed monthly. If flow measurements are found to exceed  $\pm 10\%$  of the expected flow rate, samplers are recalibrated. Flow verification transfer standard accuracy is NIST-traceable. Monthly leak checks, barometric pressure, and temperature sensor verifications will also be performed. If temperature measurements are found to exceed  $\pm 2^\circ\text{C}$  or barometric pressure sensors are found to exceed  $\pm 10\text{mmHg}$ , samplers are recalibrated. Quarterly flow, temperature, and barometric pressure audits and calibrations will be performed.

Field blanks will be provided in each batch of samples and alternate between Chukchi and Beaufort Sea monitoring sites resulting in one field blank at each site for every 6 sampling events. These samples will be handled and analyzed in the same manner as ambient samples, but do not have any sample air drawn through them. The subsequent analysis of these filters will be used to assess measurement system contamination. Analytical results from field blanks will be reported with ambient sample data. If field blanks contain confirmed detectable concentrations of target parameters, all samples handled in the field at the same time as the field blank will be flagged as suspected for possible filter handling contamination.

The sample handling and gravimetric laboratory is climate controlled for both temperature and relative humidity conditions. Temperature and relative humidity control is through a central heating/air conditioning unit. Temperature is maintained between  $20^\circ\text{C}$  and  $23^\circ\text{C}$  with an allowed twenty-four hour variation of  $\pm 2^\circ\text{C}$ . Relative humidity is maintained between 30% and 40% with an allowed twenty-four hour variation of  $\pm 5\%$ .

The temperature and relative humidity are measured and recorded in the data base with every post-weight measurement. When the temperature or relative humidity range is exceeded, analysis is discontinued until the proper range is achieved.

The mass balances are cleaned and calibrated at the beginning and upon conclusion of the work session. Periodic quality control (QC) verifications using blank and known mass standards are performed during the work session at approximately one check every 10 samples. When quality control measurements exceed the mass QC acceptance range, sample analysis is repeated. Mass standards are Class M or Class S NIST-traceable standards.

XRF systems are calibrated whenever there is a change in the physical configuration, or in the detectors, or when a problem is observed in the routine calibration checks. Thin film standards representing known concentrations of elemental species and blank film standards are used to assess system accuracy. At the beginning of each XRF session, samples from a previous session are reanalyzed with both the low current used with the QC standards and once with the high current used for normal filters. If the reanalysis values differ by more than 5%, the system is recalibrated and samples rerun. All thin film standards are NIST-traceable.

Ion chromatography systems are calibrated initially and re-calibrated whenever QC tests indicate poor system performance. Blanks and QC samples consisting of laboratory prepare matrices of known concentrations are analyzed at the beginning of every analytical batch and at least once for every 20 field samples. If the observed concentrations differ by more than 10% the problem is corrected and samples re-analyzed. Laboratory replicates and matrix spike samples are analyzed at least one for every 20 field samples.

Carbon analysis systems are calibrated initially and re-calibrated whenever QC tests indicate poor system performance. Blanks and QC samples consisting of known concentrations are analyzed at the beginning and end of every analytical batch. Replicate samples are analyzed at the rate of one per every 10 samples. Replicates often exhibit variation due to inconsistency and inhomogeneous deposit of particulate mass on the filter. Inconsistent replicates for which a reason cannot be determined will be reanalyzed.

Field blanks for all analytical procedures will be analyzed at the rate of one per sample shipment. If field blank analysis demonstrates significant concentrations of target species and elements a systemic sample handling problem is likely the cause and data will be flagged as suspicious.

## **B6 INSTRUMENT/EQUIPMENT TESTING AND INSPECTION**

### **B6.1 ACCEPTANCE TESTING**

Prior to installation, all equipment will be visually inspected to ensure no physical damage exists. After installation, and prior to the beginning of the monitoring period, samplers will be calibrated according to the procedures stated in their respective operating manuals.

Acceptance testing procedures will be documented on the appropriate calibration forms (provided in Appendix C). To ensure that the samplers continue to properly operate during the monitoring period, calibration checks of the instruments will be conducted monthly. Calibrations are performed according to manufacturer procedures using NIST-traceable flow transfer standards.

Filter media will be inspected at the laboratory. Teflon filters for Gravimetric and XRF analysis are supplied in packs of fifty. The filters are stretched Teflon® mesh filters with pore size 3.0µm, and a rigid olefin ring support. Randomly selected filters are visually inspected for unevenly stretched Teflon®, holes, and poorly attached or damaged olefin rings. Nylasorb® filters for IC analysis are supplied in packs of fifty. Filters are visually inspected for damage when prepared at the laboratory for field use. Quartz filters for TOR / TOT and ion chromatography analysis are supplied in boxes of approximately one hundred. The quantity in each box varies because filters with holes or manufacturing defects are discarded. Quartz filters are pre-fired at 900°C for at least four hours in accordance to laboratory procedures. Filter media is contained in plastic and stored in a freezer.

Random filters of each media type for each batch of samples are retained at the laboratory and analyzed as laboratory blanks to assess potential problems associated with the sampling media.

### **B6.2 SITE SURVEILLANCE AND SYSTEM CHECK PROCEDURES**

SLR station operators will inspect the condition of the monitoring equipment during each site visit. PM<sub>2.5</sub> speciation sampler flow will be verified monthly and the general station condition will be inspected to ensure that there is no environmental or vandalism damage that would impede the ability to collect valid samples. Station site technicians retrieving samples after collection will monitor equipment and station condition and notify SLR station operators of any significant observations made.

A check list will be used to document work performed during the visits and will be filed with other project field records. In the event that sampler repairs or modifications become necessary, sampler parts will be obtained from the manufacturer.

### **B6.3 PREVENTIVE MAINTENANCE AND CORRECTIVE ACTIONS**

The procedures and schedule for preventive maintenance outlined in the standard operating procedure (SOP) will be followed for this monitoring program (Appendix D). As needed, spare parts and/or equipment will be dispatched to the monitoring station for repair or sampler replacement by qualified SLR personnel.

Preventative maintenance planned for the project includes:

- Regular cleaning of down tube, cyclone and inlet surfaces;
- Replacement of denuder according to manufacturer recommendation;
- Inspect and replace o-rings as needed;
- Inspect and overhaul or replace pump and components as needed;

#### **B6.4 PM<sub>2.5</sub> PERSONNEL RESPONSIBILITIES**

Responsibilities of the PM<sub>2.5</sub> Speciation program project manager include:

- Coordination of laboratory filter shipments and sample receipt
- Coordination of field schedules;
- Ensure quality assurance data reviews according to procedure;
- Coordinate report writing and distribution; and
- Project status and budget tracking;

Responsibilities of the PM<sub>2.5</sub> Speciation quality assurance officer include:

- Receive laboratory sample results;
- Review field QA/QC documentation for adherence to project criteria;
- Provide level 2 data validation; and
- Identify and notify project manager of any field, QA/QC, or data irregularities.

Responsibilities of the PM<sub>2.5</sub> Speciation Site Operator include:

- Sampler installation and quality control verification;
- Perform monthly quality control verifications;
- Training of local site technicians;
- Coordinate sampling schedule with site technicians;
- Receive filters from the lab and load sample canisters;
- Receive exposed sample canisters from site technicians and submit to the lab; and
- Coordinate and support quarterly performance audits;

Responsibilities of the PM<sub>2.5</sub> Speciation local site technicians include:

- Receive sample canisters from site operator;
- Load canisters into the sampler and program run events;
- Prepare complete run setup and sampler statistics paperwork;
- Retrieve exposed sample canisters for short-term storage or shipment to site operator;

Responsibilities of the PM<sub>2.5</sub> Speciation data manager include:

- Receive and archive raw and validated data and QA/QC documentation;
- Prepare summary data reports according to reporting schedule;

Responsibilities of the PM<sub>2.5</sub> Speciation laboratory project manager include:

- Coordinate prepared filter shipment schedule;
- Coordinate laboratory analysis of ambient and QA/QC samples;
- Provide laboratory data validation; and
- Submit laboratory data and quality assurance documentation to the quality assurance officer for level 2 data validation.

## **B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY**

Calibration and frequency are discussed in this section. Additional information is available including instrument calibration standard operating procedures (Appendix D) and calibration worksheets (Appendix C).

### **B7.1 CALIBRATION FREQUENCY**

PM<sub>2.5</sub> speciation sampler flow, barometric pressure, and ambient temperature sensors will be calibrated according to the specified manufacturers' recommendations by SLR personnel. Field calibration is expected upon initial installation and quarterly thereafter. Monthly calibration verification checks will be performed. If calibration verifications indicate deteriorated performance, full system calibration will be performed.

### **B7.2 CALIBRATION EQUIPMENT**

All instruments used for calibration of meteorological sensors will be in current calibration, per manufactures' specifications, and traceable to National Institute of Standards and Technology (NIST) transfer standards. Calibration certifications and records will remain on file at the SLR Anchorage office. All NIST traceable equipment used for calibrations will have the relevant model number, serial number and certification date(s) recorded on accompanying forms.

## **B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES**

All spare parts inventories will be kept up to date. Each new spare part will be accepted with the same procedures mentioned in Section B6. Serial numbers for spare parts will be recorded by either the station operator or site technicians and copied to the SLR client server folder. Upon deployment of a replacement part by site technicians, the make, model, and serial number will be recorded on the appropriate form. After the repair or replacement, the instrument will be calibrated by the site technicians to ensure that it is properly functioning.

## **B9 NON-DIRECT MEASUREMENTS**

At this time SLR anticipates that the only non-direct measurements or data relevant to this project will be topography, climate history, and the surrounding land use. Any use of outside data will be quality controlled to the extent possible following QA/QC procedures outlined in this QAPP and in applicable EPA guidance documents. Refer to the approved ambient air quality and meteorological monitoring QAPPs mentioned in section A5 for discussions on non-direct measurements.



## B10 DATA MANAGEMENT

The proper management of all data associated with this project is critical to assuring the quality and usability of the monitoring results. As such, procedures will be implemented to ensure adequate data acquisition, validation, transmission, reduction, reporting, and storage of electronic and hard copy data. Summaries of the procedures to be implemented in the field and at the SLR Anchorage office for data management are provided in the subsequent sections. Overall data processes are illustrated in Figure B-9.

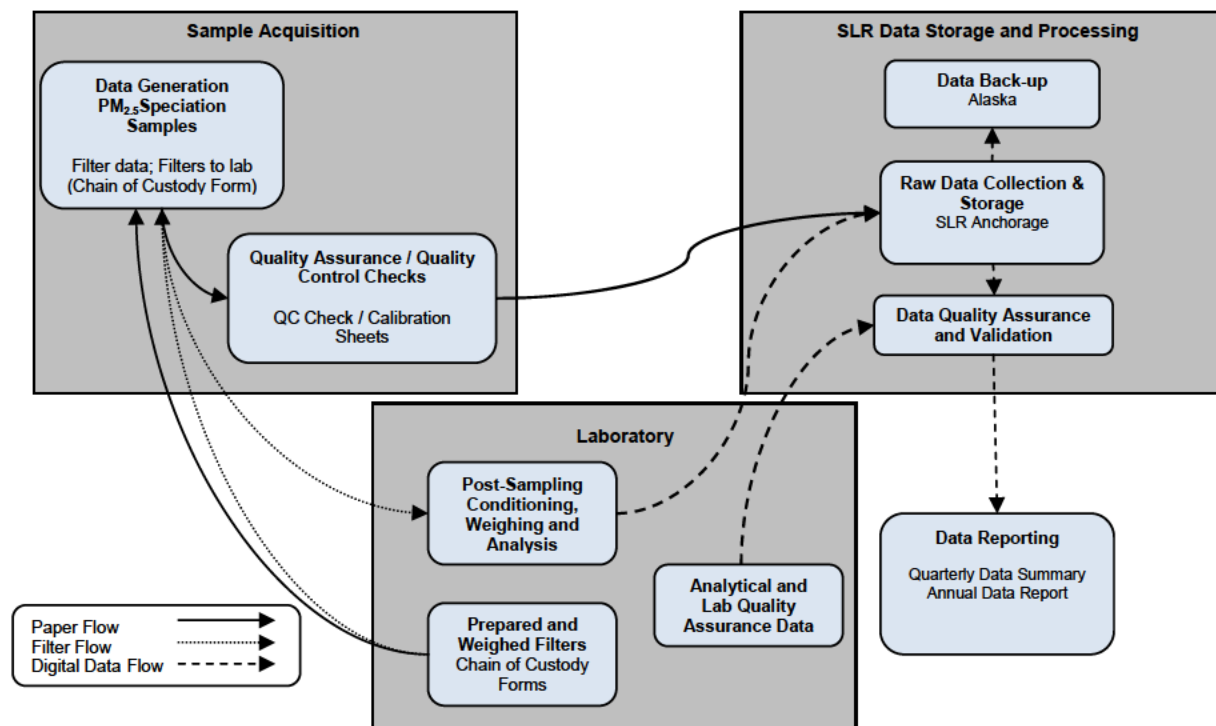


Figure B-9: PM<sub>2.5</sub> Speciation Data Processes Flow Chart

### B10.1 DATA ACQUISITION

Data acquisition will include the collection of electronic data, the generation of hand-written or typed calibration records, and the compilation of previously prepared records (e.g., NIST traceability records).

Field staff will generate electronic site visit forms and/or completed checklists. These records will be kept according to the prescribed format. Additional records pertaining to this project that have been generated by other organizations will also be collected and stored at the SLR Anchorage office as necessary.

## **B10.2 DATA VALIDATION**

During each site visit, SLR field technicians will be responsible for verifying the condition of the monitoring site. All site visits will be documented by means of a site visit memo kept with the project files. Alternately, the site visit may be documented in a designated electronic station log book. These field notes will include the following:

- Time, date, current meteorological conditions (e.g., temperature, barometric pressure, sky conditions), and personnel at the monitoring site;
- Activities completed during visit; and
- As-found and as-left observed conditions of monitoring site equipment.

Sample site visit forms can be found in Appendix C.

The data manager, or her designee, will review all hard copy records received from the field. Problems or irregularities in the records will be brought to the attention of the on-site operator for further clarification. If documentation demonstrates that the measurements were not collected within specified project quality assurance criteria, the affected data will be invalidated by the data manager and confirmed by the quality assurance officer unless compelling evidence is available to suggest it is valid. The data summary report will describe the reason for invalidating the data or if compelling evidence suggests it is valid will present that information for concurrence by regulatory authorities as needed.

Analytical data and quality assurance documentation will be reviewed by the project manager and quality assurance officer. Problems or irregularities in the records will be brought to the attention of the laboratory project manager for further clarification. If documentation demonstrates that the measurements were not collected within specified project quality assurance criteria, the affected data will be invalidated by the quality assurance officer unless compelling evidence is available to suggest it is valid. The data summary report will describe the reason for invalidating the data or if compelling evidence suggests it is valid will present that information for concurrence by regulatory authorities as needed.

## **B10.3 DATA TRANSMISSION**

Field forms, checklists, and other records will be scanned and transmitted to the SLR main office and stored in project files. Laboratory summary data will be transmitted to the SLR Anchorage office by the laboratory in standard report packages.

## **B10.4 DATA REDUCTION**

All calculations and statistical analyses will be performed using standard software packages (e.g., Microsoft Excel and WRPLOT View) that can be easily verified and audited.

## **B10.5 DATA REPORTING**

Data generated for this project will be summarized and presented in quarterly and annual data summary reports. The data summary reports will be prepared within 60 days upon completion of the monitoring period. Upon SOI's authorization, the data summary reports will be submitted to EPA for review and approval.

## **B10.6 DATA STORAGE**

All project documentation, records, data, and reports will be maintained at SLR for a period of at least five years from the completion of the project. Electronic data on the SLR servers are backed up at least once daily and will be archived at a minimum of two separate locations.

## **C. ASSESSMENTS AND OVERSIGHT**

### **C1 ASSESSMENTS AND RESPONSE ACTIONS**

Data Quality Assessments will be performed on all collected data. Data reviews will be conducted by SLR staff to determine whether the data are reasonable and representative. Data that are determined to be unreasonable or unrepresentative will be identified, flagged, and discussed in the data reports. Data reviews will include an independent record audit of field calibration records and laboratory quality control documentation. Instrument performance audits will be provided by the QA auditor on a quarterly basis. The independent record audit program will be conducted by staff independent of monitoring and laboratory operations

#### **C1.1 OPERATIONAL AUDITS**

Performance audits of the PM<sub>2.5</sub> speciation samplers will be conducted quarterly by an independent auditor. The program anticipates that independent audits will be performed by Air Monitoring Services and Technology, LLC (AMS Tech), Boulder, Colorado. Performance audits will be completed by measuring the flow rate of the sampler during its normal operating flow rate using a certified flow transfer standard. Additional checks will be completed by collocating certified temperature and barometric pressure transfer standards with the sampler to assess temperature and barometric pressure accuracy. The audit transfer standards will be provided by the station auditor and different standards will be used for audits than are used for calibration. The QA auditor will conduct the performance audits and prepare a formal audit finding report and submit it to the SLR project manager within 30 days of completion of the audit. Final audit reports will be submitted to EPA as an addendum to the quarterly data summary report. Performance audits will use the same criteria described in section B5 Quality Control. Performance audit reports will be included as supporting material in the quarterly and annual data reports.

#### **C1.2 PROJECT DOCUMENTATION ASSESSMENTS**

Field calibration records and sample shipment documentation will be examined to verify that all measurements were within expected acceptance criteria as described in section A7. Laboratory quality control documentation will be reviewed to verify that all analytical measurements were conducted in a controlled manner.

Any questioned data will be brought to the attention of the Project Manager and Quality Assurance officer who will determine whether data will be invalidated or accepted. If documentation or other information demonstrates that the measurements were not collected within specified project quality assurance criteria, the affected data will be invalidated unless compelling evidence is available to suggest it is valid. The data summary report will describe the reason for invalidating the data or if compelling evidence suggests it is valid will present that information for concurrence by regulatory authorities as needed.

### **C1.3 CORRECTIVE ACTIONS**

All deficiencies identified during operational audits or data review will be recorded and reported to the project manager within seven working days of discovery. Corrective actions to correct deficiencies will be addressed and documented in a Corrective Action Form (see Appendix C). Corrective Action Forms will include signature blocks for the recommendations(s) approval and task completion.

Any questioned data will be brought to the attention of the Project Manager and Data Manager who will determine whether data will be invalidated or accepted. Questioned data and any associated corrective actions will be discussed in the annual data report.

### **C1.4 QAPP REVISIONS**

Based on the review of the operational audit, it may be necessary to revise this QAPP. The QAPP revisions will address any project deficiencies identified in the operational audit. The project manager and permitting manager will make any necessary changes and the revised QAPP will be submitted to EPA for approval.

## **C2 REPORTS TO MANAGEMENT**

Reports to management are summarized in Table C-1.

Calibration and Sampling Event QC Verifications will be provided in the form of completed field worksheets. These worksheets will be completed and stored in the project document folder within ten days of the field activity. SLR project manager and QA officer will review the worksheets upon submission. Examples of these worksheets can be found in Appendix C.

Laboratory data summary reports will be provided by the laboratory upon completion of the lab analysis for each received filter shipment. Laboratory data summary reports will be provided to the SLR project manager within 30 days of completion of the sample analyses. Laboratory data summary reports will include:

- Sample, field blank, and laboratory blank analytical results;
- Completed chain of custody forms;
- PM<sub>2.5</sub> gravimetric laboratory environment measurements (temperature and humidity); and
- Calibration and quality control (blank, replicate and control standards) verification measurements for each analytical batch and each measurement method;

Permit conditions S.4 (Beaufort) and R.4 (Chukchi) of the air quality permits require monthly  $PM_{2.5}$  and meteorological data summaries be provided to EPA within 45 days of the end of the month. Due to the lag time associated with sample collection, shipment, laboratory analysis, and report preparation, this timeline is not practical for the  $PM_{2.5}$  speciation data. Monthly data reports to satisfy these permit conditions will come from Point Lay (Chukchi Sea) and Kaktovik (Beaufort Sea) ambient air quality and meteorological monitoring stations. These reports will contain  $PM_{2.5}$ , meteorological, and other measurements obtained from the station as well as audit reports for any performance audits performed during that period.  $PM_{2.5}$  chemical speciation data will not be submitted on a monthly basis. Quarterly data summary reports containing  $PM_{2.5}$  speciation data will be prepared and submitted to EPA within 60 days of completion of the monitoring quarter.

An annual data report will be prepared by SLR and provided to SOI and EPA within 60 days after the completion of the monitoring year. An electronic copy of the validated data in Excel spreadsheet format will be provided with the annual data report.

This annual report will include the following:

- Project Summary;
- Explanation of any variations from QAPP or other significant events
- Summaries of filter recovery and data completeness;
- Explanations for any missing, flagged, or adjusted data;
- Summaries and results of field calibrations and maintenance activities;
- Summaries of laboratory calibration and QC results;
- Data summary



Table C-1: Summary of Reports to Management

Records / Reports	Frequency	Content	Individual Responsible	Distribution
Calibration	Initial Setup	Accuracy Assessment	Field Operator	SLR Project Manager
Sampling Period QC Verifications	Before and after each monthly sampling period	Accuracy Assessment Summary	Field Operator	SLR Project Manager
Lab Data Summary	After each batch analysis	Analytical Data	Laboratory Project Manager	SLR Project Manager; Data Manager
Quarterly Summary Report <sup>1</sup>	Quarterly	Summarize data	Data Manager	SLR Project Manager, SOI, EPA
Annual Summary Report <sup>2</sup>	Annually	Summarize data	Data Manager	SLR Project Manager, SOI, EPA
Corrective Action Reports and responses	As Needed	Summarizes corrective actions taken to restore station to compliant status	SLR Project Manager	Field Operator, included in Annual Summary Report if needed

<sup>1</sup> Permit R10OCS/PSD-AK-09-01 condition R4 and R5, and Permit R10OCS/PSD-AK-2010-01 condition S4 and S5 indicate that printed monthly data summaries for PM<sub>2.5</sub> are to be submitted to EPA within 45 days of the end of the calendar month. The 45 day submittal timeline is interpreted to apply to PM<sub>2.5</sub> mass, and not the chemical speciation data. Quarterly summary reports will be submitted to EPA within 60 days of the end of the calendar quarter.

<sup>2</sup> Permit R10OCS/PSD-AK-09-01 condition R6 and R7, and Permit R10OCS/PSD-AK-2010-01 condition S6 and S7 indicate that annual data summaries and technical systems audits be submitted to EPA within 60 days of the end of the calendar year.

## **D. DATA VALIDATION AND USABILITY**

### **D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS**

#### **D1.1 DATA REVIEW REQUIREMENTS**

Data review, verification and validation are assessment techniques used to accept, reject, or qualify data in an objective and consistent manner.

Data review refers to the process that evaluates incoming data for conformance with pre-determined project criteria. During the data review process, site technicians or operators will be responsible for ensuring that:

- Field calibration and verification observations are within the operating limits of the monitoring equipment;
- Data continues to be collected and transmitted as designed; and
- Apparent outliers are identified and investigated to determine if they represent a malfunction or other issue.

Data review will be performed after each field visit to monitor instrument operations and to ensure the data have been reduced and properly formatted for further evaluation. Manual review of the data will include independent review of field documentation to identify any instrument failures.

Data package review will be performed after receipt of each analytical data package from the laboratory. Manual review of the data will include independent review of calibration and quality control documentation to identify any failures to achieve project DQOs. Manual review of the sample and field blank results will identify any abnormal low or high observations (outliers).

Any suspected data will be flagged for further evaluation and brought to the Project Manager's attention immediately. Any corrective actions will be noted on the appropriate checklists and forms.

#### **D1.1.1 Data Calculations**

Data completeness for the monitoring project will be calculated using formula D-1. The project objective for data completeness is 80 percent of the total possible number of 24 hour average samples collected for each sampling quarter. As described in section A7, samples will be considered valid if acceptable concentrations are obtained for PM<sub>2.5</sub> mass, sulfate, elemental carbon, organic carbon, and nitrate.

Data completeness ( $DC_i$ ) will be determined using equation D-1:

$$\text{D-1} \quad DC_i = S_n / S_i \times 100$$

Where:

$S_n$  = number of valid samples actually collected

$S_i$  = number of possible samples during the monitoring period.

Accuracy assessment is based on the difference between the analytical observation for a control sample and the expected known concentration of that sample. Two methods of assessment will be used by the project depending on the nature of the analytical procedure. These methods are percent difference and absolute difference.

Accuracy assessments based on percent difference ( $d_i$ ) will be determined using equation D-2:

$$\text{D-2} \quad d_i = (QC_o - QC_{tv}) / QC_{tv} \times 100$$

Where:

$QC_o$  = Observed concentration for the control sample

$QC_{tv}$  = Expected / known / true value concentration for the control sample

Assessments based on absolute difference ( $d_{abs}$ ) will be determined using equation D-3:

$$\text{D-3} \quad A_{abs} = QC_{tv} - QC_o$$

Where:

$QC_{tv}$  = Expected / known / true value concentration for the control sample

$QC_o$  = Observed concentration for the control sample

Precision assessments demonstrate the reproducibility of sample measurements. Two methods of precision assessment will be used by the project depending on the nature of the analytical procedure. These methods are relative percent difference and standard deviation of the percent difference of the control measurements.

In the case of replicate measurements, precision assessments will be based on a relative percent difference ( $d_i$ ) will be determined using equation D-4:

$$\text{D-4} \quad d_i = (X_i - Y_i) / [(X_i + Y_i) / 2] \times 100$$

Where:

$X_i$  = first concentration measurement

$Y_i$  = second / replicate concentration measurement

The standard deviation of the percent difference ( $S_j$ ) is calculated using equation D-5:

$$\text{D-5} \quad S_j = \sqrt{\frac{1}{n-1} \left[ \sum_{i=1}^n d_i^2 - \frac{1}{n} \left( \sum_{i=1}^n d_i \right)^2 \right]}$$

Where:

$n$  is the number of precision checks conducted during the monitoring period

$d_i$  is calculated using equation D-2 in the case of assessment of control standards

$d_i$  is calculated using equation D-4 in the case of assessment of replicate measurements

## **D1.2 DATA VERIFICATION AND VAILIDATION REQUIREMENTS**

Data verification refers to the process of evaluating the completeness, correctness, conformance and compliance of a specific data set against the method, procedural, or contractual specifications. The station operator will be responsible for ensuring that:

- Quality control procedures meet acceptance criteria;
- Corrective actions are documented and their effectiveness verified;
- Station log books are kept current and accurately record significant station events; and
- Method specific screening and data validation criteria are applied.

Data validation refers to a systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation is intended to confirm the degree of truth in an analytical sense.

In the data validation process, the project manager or their designee will be responsible for confirming that:

- The instruments were operated and calibrated in accordance with the manufacturers' specifications;
- The calibrations and quality control checks document that the frequency and procedural requirements defined in this QAPP were met;
- Project data completeness, precision, and accuracy goals were met; and
- The project met the specified data quality objectives as defined in this QAPP.

## D2 VALIDATION AND VERIFICATION METHODS

For the data to be considered valid, the following conditions must be satisfied:

- The instruments must each be operated and calibrated according to applicable standard operating procedure.
- Calibration records must demonstrate measurements were within acceptable criteria.
- Site visit and calibration documentation supports the validity of the data.

If any of the above are not satisfied, but alternate scientifically reasonable and compelling evidence exists that demonstrates that the data is defensible, the rationale for accepting the data will be explained in the annual data report and presented to regulators and data users for their concurrence.

### D2.1 GRAPHICAL DATA REVIEW

Microsoft Excel<sup>®</sup> or SLR's data management system will be used to calculate data completeness; minimum, maximum and average values; or other statistical assessments. This analysis is intended to identify outliers and anomalies in the data set which point to instruments operating outside of specification. Instrument diagnostic information, when available, is also graphed and compared against manufacturer operating limits.

### D2.2 STATION DOCUMENTATION

Station site visit/corrective action records, calibration records, and the data record itself are reviewed to assess the completeness of the measurement documentation. Site visit forms provide an indication of the scheduled and non-scheduled activities that took place during the monitoring project while calibration and laboratory quality control records demonstrate the outcomes of qualitative project assessments. These records should clearly demonstrate that the project quality control and quality assurance measures were met.

Table D-1 summarizes the data review processes and frequencies for the project.

**Table D-1: Data Review, Verification, and Validation Schedule**

Process	Method	Responsible Person	Frequency
Review	Data Review	Station Operator	Upon receipt of lab data package
Verification	Data Review, Records Review	Project Manager and Station Operator	Upon receipt of lab data package
Validation	Station and Laboratory Documentation	Project Manager and Data Manager	Prior to quarterly report writing

### D3 RECONCILIATION WITH USER REQUIREMENTS

The primary objective of the PM<sub>2.5</sub> Speciation Monitoring Program is to collect PM<sub>2.5</sub> chemical speciation data and comply with post construction monitoring requirements specified in permit number R10OCS/PSD-AK-09-01 (Noble Discoverer Drillship - Chukchi Sea Exploration Drilling Program) and R10OCS/PSD-AK-2010-01 (Noble Discoverer Drillship - Beaufort Sea Exploration Drilling Program). SLR will conduct an annual or final review at the completion of the monitoring project and report the results obtained during the reporting period. These reports will be compared with the established MQOs to ensure that the scope of the project has been met.

The reconciliation of data for this project will be determined by the data validation and verification process as discussed in Section D2 of this document and based upon *The Five Steps of Statistical DQA* recommended in the *Data Quality Assessment: A Reviewer's Guide* (EPA QA/G-9R):

- A review of the MQOs and sampling design will be conducted to assure data outputs from this program are still applicable. If MQOs have not been developed, MQOs will be specified before evaluating the data. A review will also be conducted of the sampling design and data collection documentation for consistency with the MQOs.
- A review of preliminary data, QA reports, basic statistics calculations, and generated graphs will be used to determine data structure and identify patterns, relationships, or potential anomalies.
- A statistical test will be selected that is most appropriate for summarizing and analyzing the data, based on the review of the MQOs, the sampling design, and the preliminary data review. Key underlying assumptions that must hold for the statistical procedures to be valid will also be identified.
- The actual data and other information about the study will be used to evaluate whether identified assumptions will hold, or whether departures are acceptable.
- Conclusions from the statistical calculations will be evaluated to determine if the monitoring design and its created data meet the required permits for the statistical test. This determination will be documented, as well as inferences drawn, as a result of these calculations. If the design is to be used again, an evaluation will be made on the performance of the sampling design.

If the preliminary data review reveals that data sets are inconsistent with the MQOs, or the underlying assumptions of the statistical test are not supported by the data and fail to meet the criteria and objectives of this monitoring project, then the project design will be reconsidered as described in this QAPP. All final decisions on data limitations will be made by EPA.